Results of Proficiency Test
Overall migration (fcm)
September 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: dr. R.G. Visser
Correctors: ing. R.J. Starink & ing. N. Boelhouwer
Report: iis14P08GM

November 2014

CONTENTS

1 INTRODUCTION

On request of a number of participants in the iis PT program it was decided to start PTs on food contact materials in 2012. This PT was repeated in 2013 and 2014.
During the contact of the food contact materials with the food, molecules can migrate from the food contact material to the food. Because of this, in many countries regulations are made to ensure food safety. The framework Regulation (EC) No. 1935/2004 applies to all food contact materials and describes a large number of requirements, e.g. limits for overall migration and specific limits for certain constituents. The determination of specific migration requires additional analytical testing following the migration step, while the determination of the overall (also called global, or total) migration requires weighing as only quantitative analytical technique.
In the iis PT on Overall Migration conducted in September 2014, 73 laboratories from 23 different countries participated (See appendix 3).
In this report, the results of the 2014 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send one sample (4 identical gloves), that gave a positive test result, labelled #14180, and to prescribe a number of test conditions (migration method, type of simulant, exposure time and temperature) to be used. Participants were also requested to report some of the test conditions that the laboratory used.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in accordance with ISO/IEC 17043:2010, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie, see also www.RVA.nl). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website www.iisnl.com.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of gloves for single use in the food industry that gave positive test results for Overall Migration was selected.
The homogeneity of the batch was checked by determination of the Overall Migration (0.5 hrs @40°C and iso octane as simulant) on 8 stratified randomly selected samples.

	Overall Migration 1st step in mg/dm^2 #14180
Sample 1	69.5
Sample 2	67.1
Sample 3	76.9
Sample 4	75.6
Sample 5	67.1
Sample 6	67.9
Sample 7	68.9
Sample 8	69.5

Table 1: results of the homogeneity test on the subsamples #14180

From the above results of the homogeneity test, the between sample standard deviation r was calculated and compared with 0.3 times the relative proficiency target standard deviations RSD_R in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Overall Migration 1st step in mg/dm^2 #14180
r(observed)	10.7
reference method	EN1186-8:2002
0.3xR(reference method)	9.94
R(reference method)	33.1
r(reference method)	15.3

Table 2: evaluation of the repeatability of the migration results on subsamples #14180

The calculated repeatability for Overall Migration on the eight samples #14180 is in good agreement with the estimated target, calculated using EN1186-8 precision data, therefore homogeneity of the samples #14180 was assumed.

To each of the participating laboratories one set of samples #14180, (4 identical gloves) was sent on September 10, 2014.

2.5 ANALYSIS

The participants were requested to determine Overall Migration on the sample using the prescribed test conditions (article filling, after gloves being turned inside out), 0.5 hrs @40°C and iso octane as simulant). It was requested to report the analytical results using the indicated units on the report form and to use a minimum number of digits and not to round the results more. It was also requested not to report 'less than' results, which are above the detection limit, because such results cannot be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also, a letter of instructions was added to the package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received.
The original data are tabulated per sample in the appendix 1 of this report.
The laboratories are represented by the code numbers.
Directly after the deadline, a reminder fax was sent to those laboratories that did not report results at that moment.
Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

The statistical calculations were performed as described in the procedures in the report ‘iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation’ of April 2014 (iis-protocol, version 3.3).

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’.
After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and or Rosner General ESD outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner General ESD test (ref. 17). Both outliers and stragglers were not included in the calculations of averages and standard deviations. Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 5; nr.14 and 15). Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study.

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8.
The z-scores were calculated in accordance with:

$$z_{(target)} = (\text{result} - \text{average of PT}) / \text{target standard deviation}$$

The $z_{(target)}$ scores are listed in the result tables in appendix 1.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used. This should be done in order to evaluate whether the reported test results are fit-for-purpose. See also appendix 5, ref. 16.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore the usual interpretation of z-scores is as follows:

$|z| < 1$ good
$1 < |z| < 2$ satisfactory
$2 < |z| < 3$ questionable
$3 < |z|$ unsatisfactory

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. Six participants reported test results after the final reporting date and seven other participants did not report any test results at all. Finally, 66 of the 73 participants submitted analysis results. These 66 laboratories reported 196 numerical test results. Observed were 8 statistically outlying results, which is 4.1%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For the determination of Overall Migration (identical to Global migration or Total Migration), the EN1186 method series (parts 1 – 15) is considered to be the official EC test method.
In this PT, given the use as mentioned in the letter of instructions, iso octane (2,2,4-Trimethylpentane) was used as simulant, cfr. EN1186 parts 1 and 14. Due to lack of a precision statement in EN1186 part 14, the target reproducibility was estimated from the reproducibility as mentioned in EN1186 part 8, annex F.

4.1 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

The calculated reproducibilities and the target reproducibilities derived from the literature standard method, here EN1186-8:02, are compared in the next table.

	unit	n	Average	2.8 * sd	R (target)
Overall migration	mg/dm^2	61	62.2	31.6	29.3

Table 3: performance overview for samples #14180

4.2 EVALUATION OF THE REPORTED TEST RESULTS

In this section the results are discussed.

residue in mg: These intermediate results were not evaluated as they are in principle dependent of the size of the contact surface used. When the article filling method is used, use of a large amount of simulant will automatically give a large contact surface and thus this will give a larger residue than use of a small amount of simulant and a small contact surface.

migration in mg/dm^2: This determination was not problematic. In total only four statistical outliers were detected, of which three results contained a calculation error. One other test result was excluded because an extremely large contact surface was reported. After rejection of the suspect data, the calculated reproducibility, is in agreement with the target reproducibility estimated from EN1186-8:02

migration in mg/kg: These test results were not evaluated as they are in principle dependent of the choice of the factor that was used for the conversion of the Overall Migration in mg/dm^2 to mg/kg. See also the discussion in paragraph 5.

4.3 EVALUATION OF THE TEST METHODS USED

The majority of the participants reported to have used a part of the EN1186 test method. Besides the general part 1 of this test method, also parts 5, 6, 8, 9 and 14 were mentioned.
The reported details of the methods that were used by the participants are listed in appendix 2.

4.4 COMPARISON WITH PREVIOUS PROFICIENCY TESTS

The number of participants increased from 46 in 2012 to 73 in this round. The percentage of outliers decreased over the years from 5.3% in 2012 to 4.1% of the numerical results in 2014.

The evolution of the uncertainty for Overall Migration in mg/dm^2 as observed in this proficiency scheme and the comparison with the findings in previous rounds is visualized in table 6.

	article filling	total immersion	EN1186
2012	18%	----	18%
2013	----	25-30%	25-30%
2014	19%	----	19%

Table 5: comparison of the uncertainties in % for Overall Migration in mg/dm^2 in the previous rounds and in the present round

5 DISCUSSION

Before the start of this PT it was clear that a wide range of test results would be reported when the choice of all test conditions would have been left to the participating laboratories. Therefore a set of predetermined test conditions was given together with the instructions to all participants.
These preset conditions were:

Sample ID	**#14180**
Simulant	Iso-octane
Exposure time	0.5 hrs
Exposure temperature	40.0 °C
Migration method	Article filling
additional	Glove turned inside out

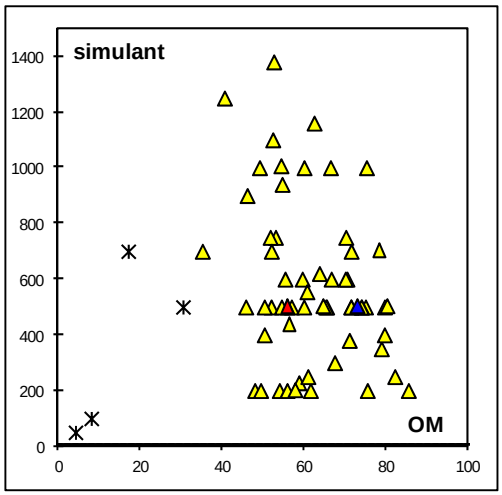
Table 4: preset test conditions used in this PT

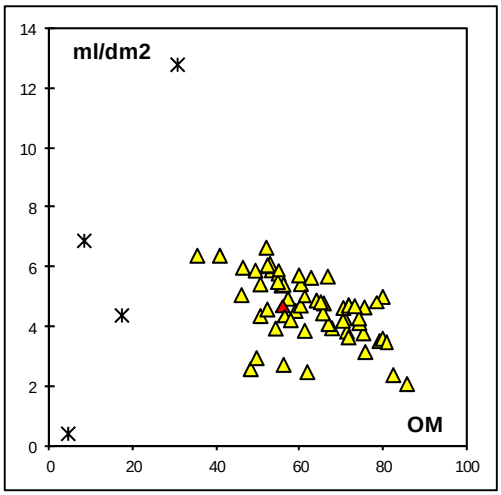
Not only a migration result was to be reported, but the participants were requested to report also the intermediate amount of residue after removal of the simulant as well as the volume of simulant used and the corresponding contact surface. Using these intermediate test results it was possible to check the calculations done by the laboratories. This revealed that several calculation errors were present. A number of laboratories corrected the calculation errors; see the original and the revised test results in appendix 1.

The amount of simulant used by each participant varied from 50 – 1380 ml, and the contact surface varied from 0.43 – 6.96 dm^2, the one extreme of 12.50 dm^2 neglected, see appendix 2. This is remarkable because in EN1186-8 as well as in EN1186-14 is mentioned that a specimen should be filled to within 0.5 cm from the top. This should lead to a large volume of simulant and

the maximum contact surface, e.g. approx 1000 ml and approx 6.5 dm^2. Hence a ratio of around 150 ml/dm^2 would be in line with the EN1186 guidelines.
In this PT, the amount of simulant used per dm^2 contact surface varied from 14.5 - 225 ml/dm^2 (see appendix 3), while on average 120 ml/dm^2 was used.

It was investigated whether the amount of simulant used was of influence on the Overall Migration (OM) test results in mg/dm^2. From the below left Youden plot it was concluded that no correlation between the amount of simulant and the overall migration is present. However, there may be a correlation between the amount of simulant used per dm^2 contact surface and the Overall Migration. In the below right Youden plot a trend from upper left to bottom right may be visible.





About two third of the laboratories reported to have evaporated all simulant and one third reported to have evaporated only part of the simulant (from 6-99%). When these two data sets were evaluated separately, only small differences were found. When only part of the simulant is evaporated, a slightly higher Overall Migration is found, while the spread of the test results is slightly small than in the case all simulant is evaporated. See the separate evaluation on page 12.

When the OM results in mg/kg were studied closely, it became clear that 25 laboratories used the conventional factor 6 to convert the result in mg/dm^2 into mg/kg (see appendix 1). A larger number of 29 laboratories did report the OM in mg/L food simulant instead. It should be noted that this does not equal mg/kg food simulant because the density of the simulant used is not 1 (one). Another 9 laboratories did use other calculation methods to calculate the OM in mg/kg or mg/L.

It is to be expected that the spread of the migration results in real life practice will be larger than observed in this PT as the test conditions like time, temperature, etc. will not be predetermined but will be selected by the individual laboratories.

Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1

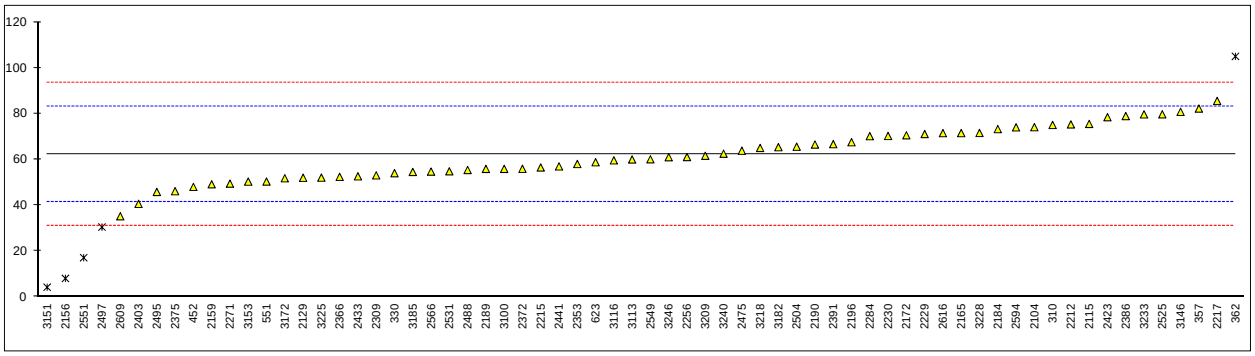
Data, statistical results and graphical results

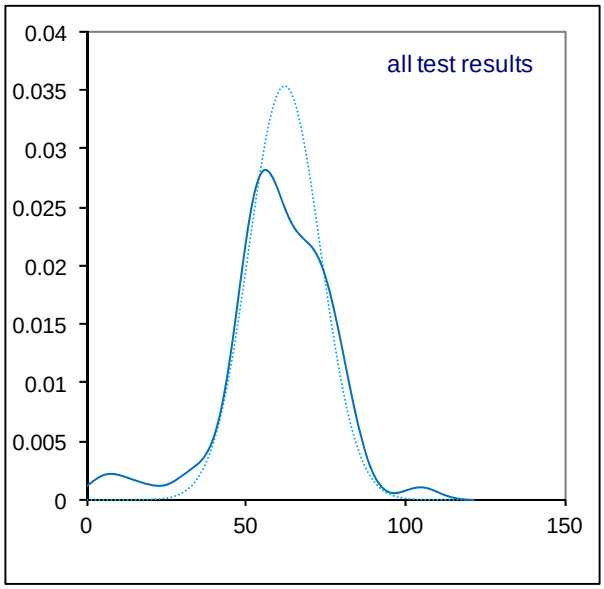
Determination of the Overall Migration on sample #14180; results in mg/dm^2

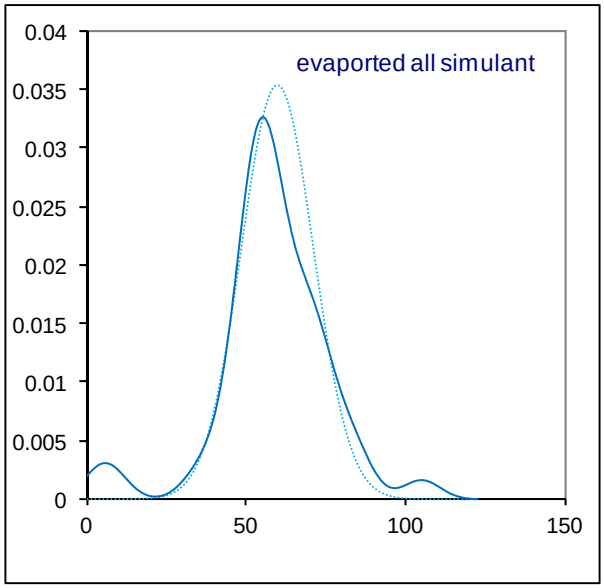
lab	method	value	mark	z(targ)	remarks
310	EN1186-6	75.05		1.23	
330	EN1186-14	54		-0.79	
357	EN1186-14	82.21		1.91	
362	EN1186-14	105	R(0.05)	4.09	
452	EN1186	48.00		-1.36	
551	EN1186-14	50.34		-1.14	
623	EN1186-14	58.77		-0.33	
632		-----		-----	
2104	EN1186-14	74.07		1.13	
2115	EN1186-8	75.50		1.27	
2129	EN1186-1	52		-0.98	
2156	in house	8.07	R(0.05), E	-5.18	iis calculated 26.96 mg/dm^2
2159	EN1186	49.16		-1.25	
2165	EN1186-1/9/14	71.5		0.89	
2172	EN1186-14	70.53		0.79	
2184	EN1186-9/14	73.23		1.05	
2186		-----		-----	
2189	EN1186-14	55.87		-0.61	
2190	EN1186-14	66.5		0.41	
2196	EN1186-8	67.51		0.51	
2212	EN1186	75.29		1.25	
2215	EN1186-1	56.50		-0.55	
2216		-----		-----	
2217	EN1186-14	85.52		2.23	
2229	EN1186-14	71.09		0.85	
2230	EN1186-1	70.24		0.77	
2241		-----		-----	
2256	EN1186-1	61.04		-0.11	
2271	EN1186-1/14	49.43		-1.22	
2284	EN1186-8	70.14		0.76	
2300		-----		-----	
2309	EN1186	53.06		-0.88	
2353	EN1186-9	58.01		-0.40	
2366	EN1186-14	52.40		-0.94	
2372	EN1186-14	55.90		-0.60	
2375	EN1186-8	46.14		-1.54	
2386	EN1186	78.92		1.60	
2391	EN1186-14	66.67		0.43	
2403	EN1186-9/14	40.6		-2.07	
2423	EN1186-14	78.42		1.55	
2433	EN1186-14	52.64		-0.92	
2441	EN1186-8	56.96		-0.50	
2475	EN1186-9	63.80		0.15	
2488	EN1186-14	55.4		-0.65	
2495	EN1186-8	45.80		-1.57	
2497	EN1186-8	30.47	ex	-3.04	used extremely large contact surface for 500 ml simulant
2504	EN1186-8	65.56		0.32	
2525	EN1186-14	79.7	C	1.67	first reported 133.37
2531	EN1186-14	54.81		-0.71	
2549	EN1186-14	60.05		-0.21	
2551	EN1186-9	17.1	C,R(0.05), E	-4.31	first reported 13.38; iis calculated 59.81 mg/dm^2
2566	EN1186-8	54.69		-0.72	
2594	EN1186-1/9/14	74		1.13	
2609	EN1186-1	35.2		-2.58	
2616	in house	71.48		0.89	
3100	EN1186-8	55.88		-0.61	
3113	EN1186-14	59.98		-0.21	
3116	EN1186-1/8/14	59.6		-0.25	
3146	EN1186-14	80.77		1.77	
3151	EN1186-5/14	4.22	R(0.05), E	-5.55	iis calculated 6.88 mg/dm^2
3153	EN1186-14	50.33		-1.14	
3172	EN1186-14	51.77		-1.00	
3182	EN1186-8	65.35		0.30	
3185	EN1186-8	54.53		-0.74	
3209	EN1186-8	61.6		-0.06	
3218	EN1186-8	65.02		0.27	
3220		-----		-----	
3225	EN1186-9	52.06		-0.97	
3228	EN1186-9/14	71.55		0.89	
3233	EUR23814 EN2009	79.67		1.67	
3237		-----		-----	
3240	EN1186-8	62.51		0.03	
3246	EN1186-8	60.98		-0.12	

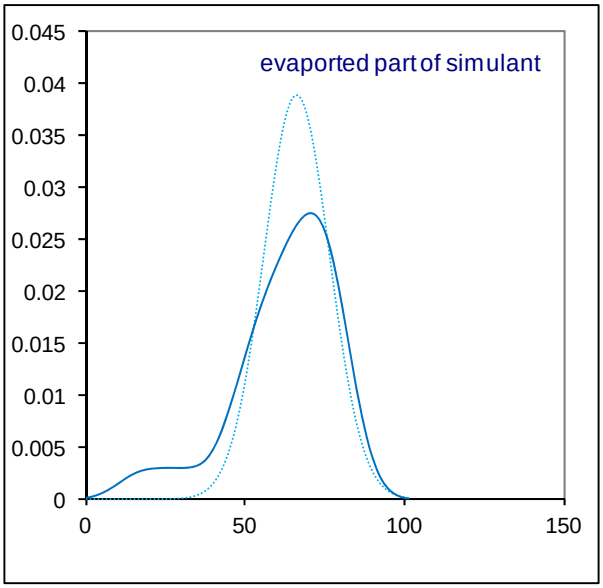
evaporated all simulant evaporated part of simulant

normality	OK		OK	OK
n	61		39	22
outliers	4	+ 1 excl.	3	1 +1 excl
mean (n)	62.218		59.888	66.348
st.dev. (n)	11.2769		11.2608	10.2923
R(calc.)	31.575		31.530	28.818
R(EN1186-8:02)	29.279		28.183	31.223





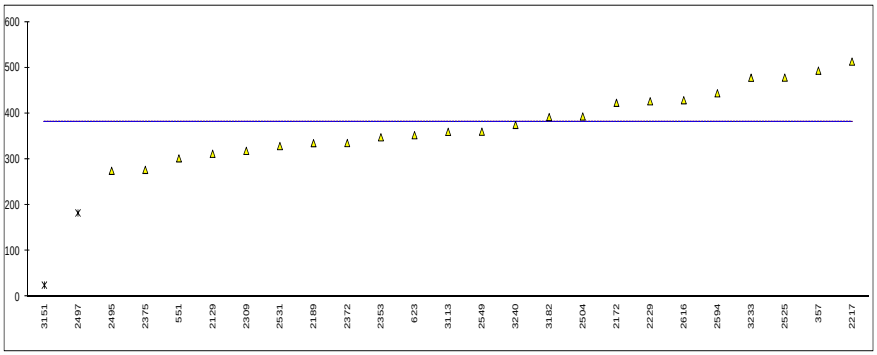


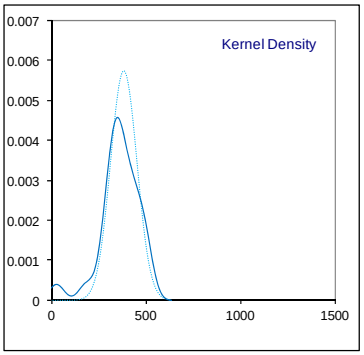


Determination of Overall Migration on sample #14180; results in mg/kg

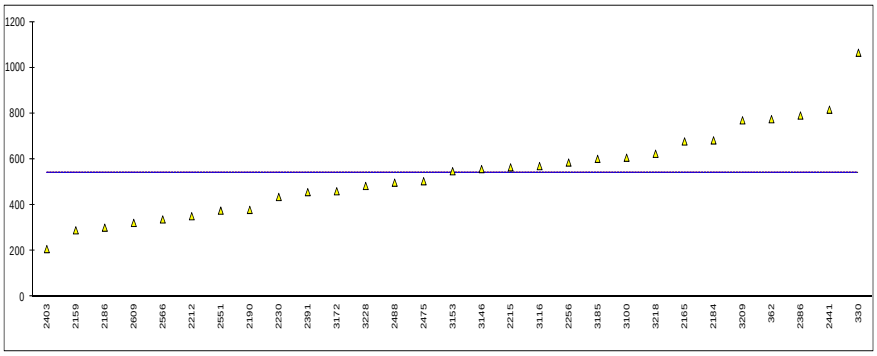
lab	method	value	mark	z(targ)	conversion factor	remarks
310	EN1186-6	37.53		-----	used factor 0.5	
330	EN1186-14	1065		-----		reported in mg/L
357	EN1186-14	493.25		-----	used conventional factor 6	
362	EN1186-14	775		-----		reported in mg/L
452		-----		-----	-----	
551	EN1186-14	302.05		-----	used conventional factor 6	
623	EN1186-14	352.6		-----	used conventional factor 6	
632		-----		-----	-----	
2104		-----		-----	-----	
2115	EN1186-8	471.70		-----		
2129	EN1186-1	312		-----	used conventional factor 6	
2156	in house	2704		-----		
2159	EN1186	290.00		-----		reported in mg/L
2165	EN1186-1/9/14	678		-----		reported in mg/L
2172	EN1186-14	423.20		-----	used conventional factor 6	
2184	EN1186-9/14	682.50		-----		reported in mg/L
2186	§64 LFBG	301.53		-----		reported in mg/L
2189	EN1186-14	335.18		-----	used conventional factor 6	
2190	EN1186-14	379.3		-----		reported in mg/L
2196	EN1186-8	536		-----		
2212	EN1186	351.63		-----		reported in mg/L
2215	EN1186-1	565.0		-----		reported in mg/L
2216		-----		-----	-----	
2217	EN1186-14	513.12		-----	used conventional factor 6	
2229	EN1186-14	426.54		-----	used conventional factor 6	
2230	EN1186-1	435.5		-----		reported in mg/L
2241		-----		-----		
2256	EN1186-1	586.00		-----		reported in mg/L
2271	EN1186-1/14	418.49		-----		
2284	EN1186-8	420.84		-----		
2300		-----		-----	-----	
2309	EN1186	318.36		-----	used conventional factor 6	
2353	EN1186-9	348.06		-----	used conventional factor 6	
2366	EN1186-14	416.51		-----		
2372	EN1186-14	335.50		-----	used conventional factor 6	
2375	EN1186-8	276.84		-----	used conventional factor 6	
2386	EN1186	790.90		-----		reported in mg/L
2391	EN1186-14	456.67		-----		reported in mg/L
2403	EN1186-9/14	208.1		-----		reported in mg/L
2423	EN1186-14	778.35		-----		
2433		-----		-----	-----	
2441	EN1186-8	816.59		-----		
2475	EN1186-9	504.30		-----		reported in mg/L
2488	EN1186-14	498		-----		reported in mg/L
2495	EN1186-8	274.80		-----	used conventional factor 6	
2497	EN1186-8	182.82		-----	used conventional factor 6	
2504	EN1186-8	393.36		-----	used conventional factor 6	
2525	EN1186-14	478.21	C	-----	used conventional factor 6	first reported 800.20
2531	EN1186-14	328.83		-----	used conventional factor 6	reported in mg/L
2549	EN1186-14	360.3		-----	used conventional factor 6	
2551	EN1186-9	375.93		-----		reported in mg/L
2566	EN1186-8	337.5		-----		reported in mg/L
2594	EN1186-1/9/14	444		-----	used conventional factor 6	
2609	EN1186-1	322.3		-----		reported in mg/L
2616	in house	428.90		-----	used conventional factor 6	
3100	EN1186-8	606.86		-----		reported in mg/L
3113	EN1186-14	359.85		-----	used conventional factor 6	
3116	EN1186-1/8/14	570.33		-----		reported in mg/L
3146	EN1186-14	557.30		-----		reported in mg/L
3151	EN1186-5/14	25.32		-----	used conventional factor 6	
3153	EN1186-14	548.00		-----		reported in mg/L
3172	EN1186-14	460.40		-----		reported in mg/L
3182	EN1186-8	392.13		-----	used conventional factor 6	
3185	EN1186-8	602.00		-----		reported in mg/L
3209	EN1186-8	770.0		-----		reported in mg/L
3218	EN1186-8	624.20		-----		reported in mg/L
3220		-----		-----	-----	
3225	EN1186-9	430.57		-----		
3228	EN1186-9/14	483.50		-----		reported in mg/L
3233	EUR23814 EN2009	478.01		-----	used conventional factor 6	
3237		-----		-----	-----	
3240	EN1186-8	375.05		-----	used conventional factor 6	
3246	EN1186-8	1.43		-----		

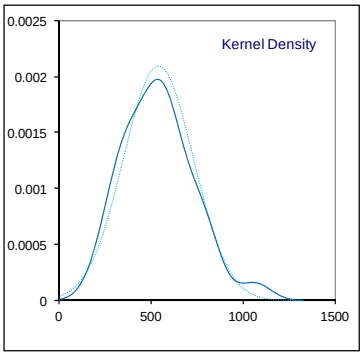
	used conventional factor 6:	data in mg/L only:
normality	OK	OK
n	23	29
outliers	2	0
mean (n)	380.441	539.391
st.dev. (n)	69.4275	190.7273
R(calc.)	194.397	534.036





Data from use of conventional factor 6 only





Data reported in mg/L food simulant

APPENDIX 2
Details reported by the participating laboratories

lab	contact surface in dm^2	volume of simulant used in ml	was all simulant evaporated ?	volume simulant evaporated in ml	Total residue after evaporation of all simulant in mg
310	3.8	500	yes	-----	285.20
330	3.96	200	yes	-----	213.4
357	2.4	250	yes	-----	197.30
362	2.20	300	yes	-----	230
452	2.6	200	yes	-----	-----
551	4.38	400	no	200	220.50
623	4.55	228	yes	-----	234.55
632	-----	-----	-----	-----	-----
2104	4.143	500	no	250	153.4 (?)
2115	3.174	200	no	approx 200	244.00
2129	4.6	500	no	50	240
2156	0.069 (m^2?)	100	yes	-----	186
2159	5.9	1000	yes	-----	290.00
2165	4.74	500	yes	-----	339
2172	4.31	600	yes	-----	304.00
2184	4.67	500	no	200	136.8 (?)
2186	6.96	1025	no	200	60.31 (?)
2189	4.70	500	yes	-----	262.57
2190	5.7	1000	no	200	75.9 (?)
2196	3.97	300	no	273	268
2212	4.67	1000	no	200	351.63
2215	4.37	437	yes	-----	246.9
2216	-----	-----	-----	-----	-----
2217	2.10	200	yes	-----	0.18 (g?)
2229	3.86	380	yes	-----	274.4
2230	4.65	750	no	200	87.1 (?)
2241	-----	-----	-----	-----	-----
2256	5.028	550	no	200	117.20 (?)
2271	2.97	200	yes	-----	146.8
2284	4.22	600	yes	-----	296.00
2300	-----	-----	-----	-----	-----
2309	5.91	750	yes	-----	313.60
2353	4.1731	198	yes	-----	242.10
2366	6.05	1100	yes	-----	317.05
2372	2.744	200	yes	-----	153.40
2375	6.0	900	no	50	15.38 (?)
2386	3.54	350	no	200	158.18 (?)
2391	4.11	600	yes	-----	274.00
2403	6.4	1250	yes	1250	260.1
2423	4.82	700	no	250	377.58
2433	6.13	1380	yes	-----	322.70
2441	4.96	500	yes	-----	282.5
2475	4.90	620	no	200	100.90 (?)
2488	5.4	600	yes	-----	299.1
2495	5.08	500	yes	-----	232.66
2497	12.8 (?)	500	no	100	390
2504	4.8	500	no	200	315.00
2525	5.02	500	no	200	400.10
2531	5.90	1000	yes	-----	328.83
2549	5.45	1000	yes	-----	327.30
2551	4.4	700	no	200	75.19 (?)
2566	5.8	940	no	200	317.25
2594	4.3	500	yes	-----	317.6
2609	6.40	700.0	yes	-----	225.6
2616	3.666	500	yes	-----	262.07
3100	5.43	500	yes	-----	303.43
3113	4.73	500	no	200	113.47 (?)
3116	5.7419	600	yes	-----	342.2
3146	3.45	500	yes	-----	278.65
3151	0.4331 (?)	50 (?)	yes	-----	2.98 (?)
3153	5.44	500	yes	-----	274.30
3172	6.67	750	yes	-----	345.30
3182	4.47	500	yes	-----	292.13
3185	5.52	500	no	200	120.40 (?)
3209	2.50	200	yes	-----	154.0
3218	4.80	500	yes	-----	312.10
3220	-----	-----	-----	-----	-----
3225	6.08	700	yes	-----	307.02
3228	4.73	700	no	200	338.45
3233	3.62	400	no	100	72.10 (?)
3237	-----	-----	-----	-----	-----
3240	5.66	1160	yes	-----	353.80
3246	3.884	250	no	150	246.25

APPENDIX 3
Details probably used by the participating laboratories

lab	Total residue after evaporation of all simulant in mg	contact surface in dm^2	volume of simulant used in ml	used ratio in ml/dm^2
310	285.20	3.8	500	132
330	213.4	3.96	200	51
357	197.30	2.4	250	104
362	230	2.20	300	136
452	-----	2.6	200	77
551	220.50	4.38	400	91
623	234.55	4.55	228	50
632	-----	-----	-----	-----
2104	306.8	4.143	500	121
2115	244.00	3.174	200	63
2129	240	4.6	500	109
2156	186	6.9	100	14.5
2159	290.00	5.9	1000	169
2165	339	4.74	500	105
2172	304.00	4.31	600	139
2184	342	4.67	500	107
2186	309.089	6.96	1025	147
2189	262.57	4.70	500	106
2190	379.5	5.7	1000	175
2196	268	3.97	300	76
2212	351.63	4.67	1000	214
2215	246.9	4.37	437	100
2216	-----	-----	-----	-----
2217	180	2.10	200	95
2229	274.4	3.86	380	98
2230	326.6	4.65	750	161
2241	-----	-----	-----	-----
2256	322.3	5.028	550	109
2271	146.8	2.97	200	67
2284	296.00	4.22	600	142
2300	-----	-----	-----	-----
2309	313.60	5.91	750	127
2353	242.10	4.1731	198	47
2366	317.05	6.05	1100	182
2372	153.40	2.744	200	73
2375	276.84	6.0	900	150
2386	276.82	3.54	350	99
2391	274.00	4.11	600	146
2403	260.1	6.4	1250	195
2423	377.58	4.82	700	145
2433	322.70	6.13	1380	225
2441	282.5	4.96	500	101
2475	312.79	4.90	620	127
2488	299.1	5.4	600	111
2495	232.66	5.08	500	98
2497	390	12.8 (?)	500	39
2504	315.00	4.8	500	104
2525	400.10	5.02	500	100
2531	328.83	5.90	1000	169
2549	327.30	5.45	1000	183
2551	263.15	4.4	700	159
2566	317.25	5.8	940	162
2594	317.6	4.3	500	116
2609	225.6	6.40	700.0	109
2616	262.07	3.666	500	136
3100	303.43	5.43	500	92
3113	283.675	4.73	500	106
3116	342.2	5.7419	600	104
3146	278.65	3.45	500	145
3151	2.98	0.4331 (?)	50 (?)	115
3153	274.30	5.44	500	92
3172	345.30	6.67	750	112
3182	292.13	4.47	500	112
3185	301	5.52	500	91
3209	154.0	2.50	200	80
3218	312.10	4.80	500	104
3220	-----	-----	-----	-----
3225	307.02	6.08	700	115
3228	338.45	4.73	700	148
3233	288.4	3.62	400	110
3237	-----	-----	-----	-----
3240	353.80	5.66	1160	205
3246	246.25	3.884	250	64

APPENDIX 4

Number of participating laboratories per country

1 lab in	BRAZIL
1 lab in	BULGARIA
1 lab in	DENMARK
1 lab in	FINLAND
4 labs in	FRANCE
6 labs in	GERMANY
7 labs in	HONG KONG
1 lab in	HUNGARY
5 labs in	INDIA
1 lab in	INDONESIA
6 labs in	ITALY
2 labs in	MALAYSIA
21 labs in	P.R. of CHINA
2 labs in	PHILIPPINES
1 lab in	SAUDI ARABIA
1 lab in	SERBIA
2 labs in	TAIWAN R.O.C.
2 labs in	THAILAND
1 lab in	THE NETHERLANDS
4 labs in	TURKEY
1 lab in	U.S.A.
1 lab in	UNITED KINGDOM
1 lab in	VIETNAM

APPENDIX 5

Abbreviations:

C = final result after checking of first reported suspect result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner outlier test
R(0.05) = straggler in Rosner outlier test
n.a. = not applicable
E = possible calculation error
W = result was withdrawn

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
2 EN 1186-1:02 - Guide to the selection of conditions and test methods for overall migration
3 EN 1186-8:02 - Test methods for overall migration into olive oil by article filling
4 EN 1186-14:02 - Test methods for 'substitute tests' for overall migration from plastics intended to come into contact with fatty foodstuffs using test media iso-octane and 95 % ethanol
5 ASTM E1301-03
6 ISO 5725-86
7 ISO 5725, parts 1-6, 1994
8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
10 IP 367/96
11 DIN 38402 T41/42
12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
13 J.N. Miller, Analyst, 118, 455, (1993)
14 Analytical Methods Committee Technical Brief, No4 January 2001
15 The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)
16 R.G. Visser, Reliability of proficiency test results for metals and phthalates in plastics, Accred Qual Assur, 14:29-34 (2009)
17 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), pp. 165-172, (1983)